UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: December 30, 1986

SUBJECT: Performance Audit - O'Brien & Geere Laboratory, Syracuse, New York - National Lead (NL) Granite City, Illinois Site

FROM: James H. Adams, Jr., Chief
Quality Assurance Office

TO: Norman Niedergang, Chief
CERCLA Enforcement Section

ATTENTION: Brad Bradley

The Quality Assurance Office, on August 29, 1986, provided unknown reference samples (Performance Evaluation samples) to the O'Brien & Geere Laboratory, Syracuse, New York. These were to be tested for 8 metals by graphite furnace atomic absorption spectroscopy as specified by the Quality Assurance Project Plan (QAPP) for the NL - Granite City, Illinois remedial investigation. Results for these samples were received by our Office on December 12, 1986.

David A. Payne, Chemist, of our Office evaluated these results on December 23, 1986, and orally transmitted our major recommendations to Mr. Bradley on this same day.

Attached are tabulated results for the reference samples. Experimental results are accurate. The laboratory is to be complimented for this over-all accuracy. The laboratory should be considered acceptable for the graphite furnace determinations specified by the QAPP with the provision that above reference samples did not contain the interferences expected of certain groundwaters at the site. The reference sample results and supporting documentation indicate the atomic absorption system is accurately calibrated and is operated in appropriate metals concentration ranges required by the QAPP. Analysis of actual groundwaters will undoubtedly require method of standard addition estimates and calculations that are unnecessary for the Performance Evaluation samples fabricated in reagent water.

We offer a few comments on the experimental records provided with the Performance Evaluation results. These were discussed with David Hill of OBG Laboratories by telephone on December 23, 1986.

1. All sample aliquots tested by graphite furnace were first digested using RCRA Method 3020. The QAPP does not require digestion of groundwaters (except a few lead determinations). Antimony was probably digested with nitric acid when it should have used a final digestion of hydrochloric acid.

2. We recommend that OBG Laboratories identifies specific laboratory digestion blanks with a unique sample number instead of the noted "MB". See "Sample Preparation and Digestion" record sheet.

3. The instrument response (peak absorbance or integrated absorbance) should be tabulated for each calibration standard, control, blank, sample aliquot, etc., on each "Atomic Absorption Injection Logbook" record. Calculated concentrations are reported.

4. OBG should be careful of the negative blank values calculated by the graphite furnace data system. Reported negative blank values were all within plus or minus the QAPP required detection limits. Significant negative blank values was present for chromium. Negative blank values were also exhibited by nickel and selenium.

5. The instrument response for graphite determinations of cadmium appears too sensitive for the concentration range of interest. Absorbance peak height values greater than 0.5 and 1.0 are recorded for standards. The same is true for samples or spiked samples. Cadmium is such a sensitive elemental analysis by graphite furnace that it is often necessary to provide sample dilution prior to analysis. OBG should check their instrument operation manual to see if absorbance value for cadmium standard curves and samples shouldn't be maintained at 0.4 or less. This may require additional sample dilutions and more dilute standards. Please note that the peak shape of standards are slightly different than that for samples. Could this be due to the absorbance ranges being tested? It would be an excellent idea for the "Atomic Absorption Injection Log Book" to include records of volume injected for samples, standards, etc.

6. We note that a negative bias between 10% and 20% is present in cadmium results and cadmium control samples (2.44 ug/l versus 2.70 ug/l true value). The observed bias provides for a marginally "acceptable" performance evaluation between 1 and 10 ug/l concentration. The reason for the bias should be found and corrected. The bias is significant when determining cadmium concentrations at specific concentration levels of interest for the study. Please note that 7 elements were determined quite accurately and 1 with borderline acceptability. This is excellent performance for any laboratory.

cc: D. Payne, QAO
S. Hong, CES
M. Gade, WMD

PERFORMANCE EVALUATION RESULTS
FOR GRAPHITE FURNACE DETERMINATIONS
BY OBG LABORATORIES, SYRACUSE, NEW YORK

ELEMENT	SAMPLE NUMBER	REQUIRED DETECTION LIMIT* (ug/l)	REPORTED RESULT (ug/l)	EXPERIMENTAL RESULT** (ug/l)	TRUE VALUE*** (ug/l)	PERFORMANCE EVALUATION
Antimony	3	20	20		16.9	ACCEPTABLE
	4	20	59		51	ACCEPTABLE
Arsenic	1	5	24		24	ACCEPTABLE
	2	5	52.4		60	ACCEPTABLE
Cadmium	1	1	3.37		4	ACCEPTABLE[a.]
	2	1	8.24		10	ACCEPTABLE[a.]
Chromium	1	5	<5	4.2	6	ACCEPTABLE
	2	5	13.1		14	ACCEPTABLE
Copper	1	10	<10	8.8	10	ACCEPTABLE
	2	10	17.9		18	ACCEPTABLE
Lead	1	5	9.6		8	ACCEPTABLE
	2	5	19.1		18	ACCEPTABLE
Nickel	1	10	<10	7.6	8	ACCEPTABLE
	2	10	12.8		16	ACCEPTABLE
Selenium	1	2	3.7		4	ACCEPTABLE
	2	2	9.4		10	ACCEPTABLE

* - Detection Limit from QAPP
** - Experimental Values taken from Atomic Absorption Injection Logbook records attached to Performance Evaluation
*** - Provided by EMSL-Cincinnati, EPA
a. - Negative 15% bias present in all results and controls tested. This bias should be eliminated.

OBG LABORATORIES, INC.

QUALITY ASSURANCE BRANCH

DEC 12 1986

ENVIRONMENT SERVICES DIVISION

Laboratory Report

CLIENT NL FURNACE PERFORMANCE EVALUATION JOB NO. 2488.012.517

DESCRIPTION Results reported as ug/l (ppb)

DATE COLLECTED _____ DATE REC'D. _____ DATE ANALYZED _____

	A5880 NL#1	A5881 NL#2	A3122 NL#3	A3124 NL#4	REQUIRED DETECTION LIMIT
ANTIMONY	-	-	20.0	58.9	20.
ARSENIC	24.0	52.4	-	-	5.
CADMIUM	3.37	8.24	-	-	1.
CHROMIUM	<5.	13.1	-	-	5.
COPPER	<10.	17.9	-	-	10.
LEAD	9.6	19.1	-	-	5.
NICKEL	<10.	12.8	-	-	10.
SELENIUM	3.7	9.4	-	-	2.

Methodology: Federal Register — 40 CFR, Part 136, October 26, 1984

Units: mg/l (ppm) unless otherwise noted

Comments:

Authorized: _____

Date: December 11, 1986

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